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(54) Method for performing chemical reactions utilisable in the manufacture of phenol formaldehyde resins

(57) A chemical reaction, for example the reaction of phenol and formaldehyde components in the manufacture of phenol formaldehyde resin, is performed by feeding reactants along a narrow channel (9) defined between the inner surface of a tube (1) and the outer surface of a rod (3) which is rotated coaxially within the tube. In alternative arrangements the tube rotates and the rod is fixed, or both the tube and rod rotate.

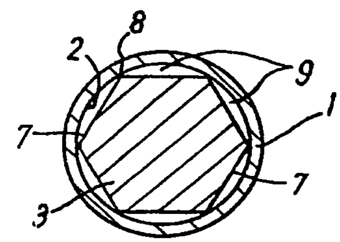

FIG.2

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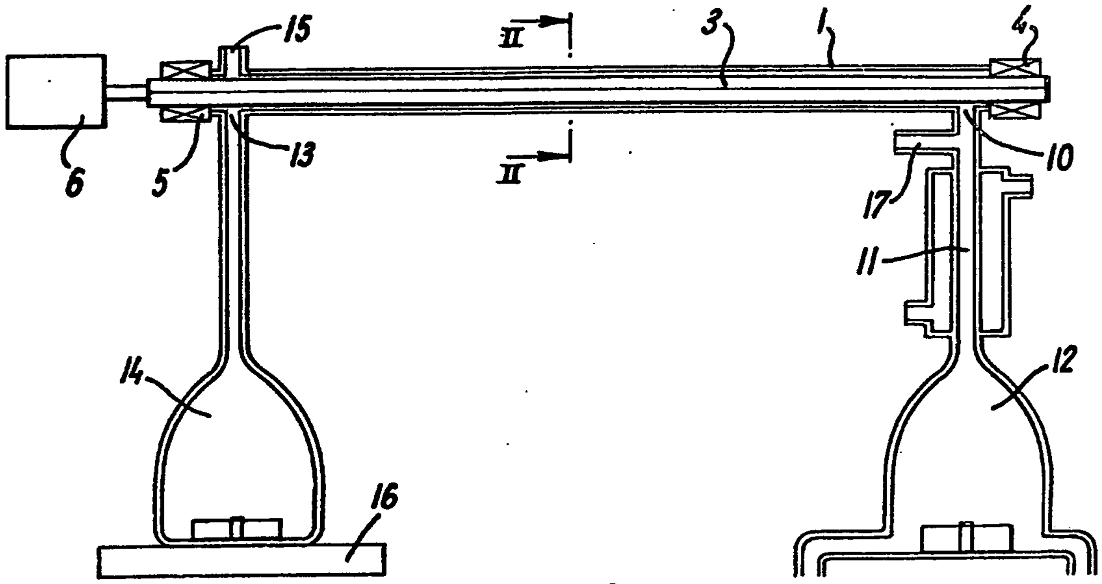

FIG.1

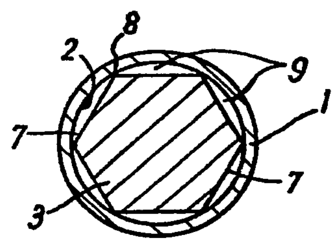

FIG.2

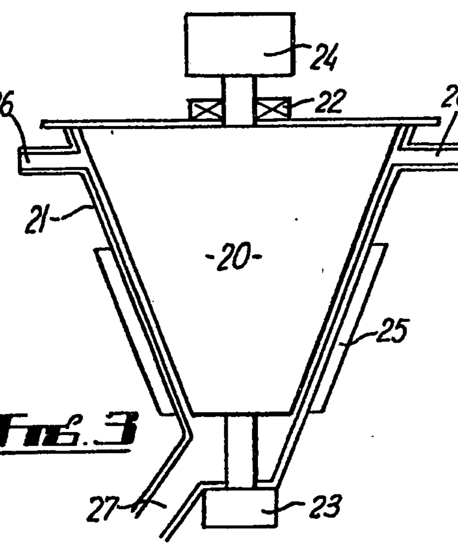

FIG.3

SPECIFICATION

Method for performing chemical reactions utilisable for example in the manufacture of phenol formaldehyde resins

This invention relates to a method for performing chemical reactions and is particularly although not exclusively concerned with the manufacture of phenol formaldehyde resins.

In the manufacture of phenol formaldehyde resins it is known to use a batch process involving the reaction of phenol and formaldehyde components in the presence of a catalyst with stirring in a reaction vessel. However, this known procedure may be disadvantageous in that: the required batch apparatus may be bulky, inconvenient and expensive; and the progress of the reaction may be difficult to control, especially in the case of the highly exothermic acid catalysed novolak reaction, whereby efficient production of large quantities of resin of uniform satisfactory properties may be difficult to achieve, and there is even the danger that the reaction may run away and result in explosion.

An object of the present invention is to provide a reaction procedure applicable to the manufacture of phenol formaldehyde resin and with which the abovementioned disadvantages can be avoided or at least appeciably mitigated.

According to one aspect of the present invention therefore there is provided a method of performing a chemical reaction wherein reactants are fed along and react within at least one channel defined between the inner surface of a tube and the closely adjacent outer surface of an elongate member which extends longitudinally within said tube, whilst one of said tube and elongate member is rotated relative to the other.

The invention also provides apparatus for use in performing the above method, said apparatus comprising a tube, an elongate member extending longitudinally within said tube, at least one channel being defined between the inner surface of the tube and the outer surface of the elongate member, means for feeding reactants along the said channel or channels between an inlet and an outlet in communication with opposite ends thereof, and means for rotating one of said tube and said member relative to the other about its longitudinal axis. In a preferred embodiment, the tube is fixed and the elongate member is axially rotatable although it is also possible to use an arrangement in which the tube rotates and the member is fixed or in which both rotate.

The method and apparatus of the invention are particularly suited to the performance of chemical reactions on a continuous basis and can enable large quantities of reaction product to be obtained with equipment of relatively simple, convenient and inexpensive form. Moreover, in so far as the quantity of material involved in the reaction at any time can be kept at a low level, corresponding to the quantity of material which can be contained within the said channel or channels, it is possible to maintain good control of the progress of the reaction whereby efficient manufacture of reaction product of uniform satisfactory properties can be achieved. Also, the danger of exothermic reactions running away and resulting in explosions can be avoided or at least much reduced.

It is visualised that the invention will find particular application in the context of the manufacture of phenol formaldehyde resins, especially utilising the acid catalysed, highly exothermic novolak reaction. It is however, to be understood that the invention is not intended to be restricted to this context and instead may be applied to the alkali catalysed resole reaction, or to the manufacture of any other suitable resin or indeed to any other suitable chemical reaction.

Most preferably the relative shapes and sizes of the tube and elongate member are such that the space therebetween, which defines the said channel or channels, is appropriate to the formation of a thin film of the reaction mixture. With such thin film it is possible to achieve especially good mixing of reactants and uniformity of reaction conditions (particularly temperature distribution) throughout the mixture. Also, this arrangement particularly facilitates careful control of reaction conditions to the extent that any changes or adjustments thereto (even including arrest of the reaction) can be implemented quickly and conveniently as and when required. If desired monitoring means may be provided to monitor one or more reaction parameters such as temperature and/or pressure, whereby for example fall or rise in temperature (indicating for example undesired reduction in or acceleration of reaction rate) or rise in pressure (indicating for example undesired gellation or solidification of the reaction mixture) can be detected thereby enabling appropriate remedial action to be taken when necessary.

Preferably also, at least one projection is provided on the said outer surface of the said elongate member, or the inner surface of the tube, to scrape or sweep the reaction mixture around the inside of the tube during said rotation, such scraping or sweeping action facilitating rapid, even flow and uniform mixing of the reactants. Said projection may take any suitable form but preferably comprises a ridge or edge running longitudinally. Where there are two or more said channels one or more said longitudinal projections may be provided at a boundary between circumferentially adjacent said channels. The or each said longitudinal projection is preferably straight and parallel to the axis of rotation, such arrangement facilitating rapid flow of the reaction mixture through the channel or channels and avoiding accumulation of pockets or plugs of such mixture within same, although if desired and as appropriate, longitudinal projections which are not straight (being for example curved) may also be used.

The size and shape of the or each said channel may remain constant during said rotation. Alternatively, if desired, the size and shape may

change for example due to a regular or irregular fluctuation in the location of the axis of rotation of the tube or member (which may be caused by permitted play in rotary bearings supporting same or due to appropriate shaping of the confronting surfaces of the member and tube and appropriate location of the said rotational axis.

A permanent clearance may be maintained between all parts of the inner surface of the tube and the elongate member. Alternatively, the member may contact the tube surface during at least part of the rotational cycle. Such contact may occur between the tube surface and the aforesaid projection or projections. Where such contact occurs, one or both of the inner surfaces of the tube and elongate member may be appropriately formed or covered or treated to facilitate smooth sliding movement therebetween.

The said inner surface of the tube may be circular in cross-section as also may be the outer surface of the elongate member and in accordance with one embodiment such circular tube and member may be arranged coaxially. It is however also possible to use non-circular constructions. Thus, in one preferred embodiment the outer surface of the elongate member and/or the inner surface of the tube has multiple faces thereto, which faces may be curved or flat as desired; and, in a particularly preferred embodiment, the member has multiple like flat faces and is of regular polygonal (especially hexagonal) cross-section. The cross-sectional form of the confronting tube and member surfaces may be uniform in shape and size along the length thereof. Alternatively however, the cross-sectional form of either or both of the tube and member may change longitudinally. For example, the tube and member may be of a conical or tapered construction. Similarly, the or each channel cross-section may be uniform or non-uniform longitudinally as desired.

Any suitable material or combination of materials may be used for the tube and member including glass, stainless steel and other materials.

Means may be provided for heating or cooling the tube and/or the elongate member as desired. Such heating or cooling may be applied uniformly or non-uniformly longitudinally.

The reactants (and any other ingredients such as catalysts, solvents and the like) may be fed to the channel or channels through said inlet at one end of the said tube and the reacted mixture may be removed through said outlet at the opposite end of the tube. Alternatively or additionally said inlet and/or said outlet may be provided at a side position on the tube.

Provision may be made for controlling the pressure of the reactants within the or each channel. Thus the or each channel may be sealed except for the inlet and outlet openings thereto and these openings may be attached to connections appropriate to the maintenance of a desired pressure within the or each channel. Also, the or each channel may be shaped as for example by a constriction or tapering along the length thereof to give an abrupt or progressive pressure change.

The reactants (any any other ingredients) may be fed to the inlet with one or more metering pumps and a mixing chamber may be interposed between such pump or pumps and the inlet. There may also be a pre-heater section before the inlet. The outlet may be vented and may discharge to a receiver which may be heated and may contain a stirrer.

The tube and member may be arranged to give a predetermined disposition for said channel or channels between the inlet and outlet. Thus, the or each channel may be inclined at an angle set to give a desired residence time having regard to parameters such as product viscosity.

The elongate member and tube may be rotatably mounted one relative to the other in any suitable manner. Thus the member may project beyond the tube at each end and such projecting ends may be mounted in respective bearings. Alternatively one or both ends of the member may be rotatably mounted in bearings within the tube. It is also possible to mount one end only in bearings inside or outside the tube, the other end being unsupported within the tube. Any suitable form of drive motor may be used for effecting rotation. Rotation may be at a fixed or variable speea.

The tube and/or member may be formed in detachable longitudinal sections to facilitate dismantling for example to free blockages. Such sections may be insulated relative to each other to help establish different temperature zones.

The invention will now be described further by way of example only and with reference to the accompanying drawings in which:—

Fig. 1 is a diagrammatic longitudinal sectional view of one form of apparatus according to the invention;

Fig. 2 is a section on the line II—II of Fig. 1; and

Fig. 3 is a diagrammatic sectional view of an alternative embodiment.

Fig. 1 shows a chemical reactor comprising a cylindrical stainless steel tube 1 of length 71 cm and internal diameter 0.8 cm. The tube 1 has a thin wall of uniform thickness throughout its length and has a smooth, continuous, imperforate, cylindrical inner surface 2. A solid stainless steel rod 3 extends along the tube 1 and projects beyond the tube at each end thereof. Each projecting end of the rod is supported in bearings 4, 5 such that the rod is free to rotate about its longitudinal central axis which is coaxial with the tube axis, said bearings sealing the tube ends. A motor 6 is drivably connected to one end of the rod 3 through the respective bearing 5.

As shown in Fig. 2, the rod 3 is of regular hexagonal cross-section and has six like flat faces 7 on its outer surface with six straight edges 8 between same, such edges 8 extending parallel to the axis of the rod. The edges 8 may make sliding

contact with the inside of the tube or alternatively a slight clearance may be maintained between same. Six like channels 9 extending throughout the entire length of the tube 1 are defined between the inner surface 2 of the tube 1 and the six flat faces 7 of the rod 3, such channels 9 being straight and of narrow elongate cross-section.

At one end the tube has an inlet fitting 10 which is connected via a pre-heater section 11 to a mixing chamber 12, such pre-heater section 11 comprising a short feed tube (say 75 mm long) enclosed within a jacket through which steam or hot water can be circulated. The mixing chamber 12 contains a stirrer and is connected to two metering pumps (not shown) respectively connected to a source of mixed formaldehyde and phenol (say 87% strength paraformaldehyde with phenol crystals giving a ratio of 1 mole phenol: 0.85 mole formaldehyde) and to a source of acid catalyst (say aqueous sulphamic acid). The inlet fitting 10 is also connected (at 17) to a pressure gauge (not shown).

The opposite end of the tube 1 has an outlet fitting 13 which leads to a conical receiver 14 and a vent outlet 15, such receiver 14 being provided with a stirrer and also a heater 16 (for example a hot plate beneath the receiver).

The body of the tube 1 is provided with a heating system (not shown) comprising for example an electrical heating tape wrapped around the tube.

The tube 1 may be positioned horizontally or at a slight angle to the horizontal with the outlet end below the inlet end.

In use, the phenol/formaldehyde mixture and the catalyst are fed to the mixing chamber 12 (say at rates of 305 ml/hr for the formaldehyde/phenol and 57.5 ml/hr for the catalyst) and from there pass to the inlet end of the tube 1 via the pre-heater section 11. Such reaction mixture then flows along the channels 9 defined between the rod 3 and the tube 1 to the outlet end of the tube 1 whilst the rod 3 is rotated with the motor 6.

The reaction mixture is contained as a thin film within the channels 9 and such thin film is swept around the inside of the tube 1 by the edges 8 as the rod rotates. The amount of material within the tube at any time may be of the order of 8 ml and the residence time may be 1.33 mins/ml.

The phenol and formaldehyde components react within the tube, such reaction being initiated, if desired, by heating the body of the tube using the abovementioned tube heater. After such initiation, heating may be continued or, alternatively, the reaction may proceed without external heating of the tube as a self-sustaining exothermic reaction. The resulting reacted mixture is expelled from the outlet end of the tube into the receiver 14 from where the final product, i.e. phenol formaldehyde resin is recovered. The temperature within the tube may be 120°C and the receiver may be heated to 160°C.

With this arrangement, due to the use of a thin film reaction mixture which is continuously agitated by the action of the rotating hexagonal rod, it is possible to ensure homogeneous mixing of the reactants and uniform temperature distribution throughout same, maintenance of such uniform temperature distribution also being assisted by conduction through the stainless steel rod and tube. Accordingly, the reaction can proceed in a carefully controlled manner such as to give a good quality resin product. Moreover, the process can be operated continuously for several hours or longer whilst maintaining uniformity of produced resin properties. Run-away exothermic reaction and hence the danger of explosion can be avoided, as also can blocking of the tube due to gellation therein. Further, having regard to the small amount of reaction mixture contained within the reactor at any time, any required adjustments to reaction conditions (for example, temperature, overall or relative feed rates or the like), or even arrest of reaction, can be implemented rapidly. Monitoring devices, such as the pressure gauge connected at 17, can be used to detect adverse tendencies (such as a pressure increase due to incipient gel formation in the tube) so that remedial adjustment of reaction conditions can be effected when necessary.

Since it is possible accurately and reliably to achieve and maintain desired reaction conditions it will be understood that efficient and economic resin production can be attained.

The method described above is suited to the efficient reliable and safe production of large quantities of resin. Further, the required apparatus can be of a particularly simple, convenient and inexpensive form and is suited to use in a wide range of different contexts including on-site applications for example in the building industry and also for chipboard manufacture.

It is of course to be understood that the invention is not intended to be restricted to the details of the above embodiment which are described by way of example only. Thus for example, the invention is not restricted to the production of phenol formaldehyde resin by reaction of paraformaldehyde with phenol catalysed by sulphamic acid. The invention can also be applied to the reaction of formaldehyde with phenol as catalysed by N/10 hydrochloric acid or indeed to any other suitable reaction.

Further, the inner surface of the tube need not be wholly smooth but may be rifled or otherwise formed to promote transfer of materials along the tube. Moreover, if desired provision may be made for the addition of materials at one or more positions along the length of the tube and/or for the removal of by-products or excess reactants for example solvents or distillates. It is also possible to allow for the recycling of products from one zone of the tube to another.

The outlet end of the tube may communicate with appropriate finishing stages, for example for flaking or pelleting.

Additionally or alternatively to the use of pumps for reactant feed purposes it is possible to use an archimedian screw device which may be defined by an end portion of the rod or which may

be used in tandem with the rod. It is also possible to use other feed devices for example a constant head device and an orifiçe plate obviating any pump.

As mentioned, the tube 1 and rod 3 need not be horizontal as shown in the drawing but instead may be inclined to the horizontal. By selection of the inclination it is possible to attain a desired residence time having regard to other parameters such as the product viscosity.

Instead of using the separate mixing chamber 12, it is possible to provide an integral mixing section at an inlet end of the tube to which ingredients are fed separately.

With the embodiment of Fig. 3, a conical member 20 is rotatably mounted within a conical outer tube 21. The member 20 is mounted on bearings 22, 23 and is connected to a motor 24. The outer tube is provided with an electrical heating jacket 25 and also with top feed inlets 26 and a bottom feed outlet 27. With this arrangement it is possible to generate a desired pressure increase due to the tapered form of the gap between the member 20 and the tube 21. The member and tube may be arranged with their axes vertical, or inclined to the vertical as desired.

Claims

1. A method of performing a chemical reaction wherein reactants are fed along and react within at least one channel defined between the inner surface of a tube and the closely adjacent outer surface of an elongate member which extends longitudinally within said tube, whilst one of said tube and elongate member is rotated relative to the other.

2. A method according to claim 1, applied to the manufacture of resin.

3. A method according to claim 2, applied to the manufacture of a phenol formaldehyde resin.

4. A method according to any one of claims 1 to 3, wherein said reactants are premixed before feeding along the or each said channel.

5. Apparatus for use in performing the method of any one of claims 1 to 4 comprising a tube, an elongate member extending longitudinally within said tube, at least one channel being defined between the inner surface of the tube and the outer surface of the elongate member, means for feeding reactants along the said channel or channels between an inlet and outlet in communication with opposite ends thereof, and means for rotating one of said tube and said member relative to the other about its longitudinal axis.

6. Apparatus according to claim 5, wherein the tube is fixed and the elongate member is axially rotatable.

7. Apparatus according to claim 5 or 6, including monitoring means for monitoring one or more reaction parameters.

8. Apparatus according to any one of claims 5 to 7, wherein at least one projection is provided on one of said inner and outer surfaces.

9. Apparatus according to claim 8, wherein the or each said projection is provided on said outer surface of said member.

10. Apparatus according to claim 8 or 9, wherein the or each projection comprises a longitudinally extending ridge or edge.

11. Apparatus according to claim 10, wherein there are two or more channels and one or more said longitudinal projections is provided at a boundary between circumferentially adjacent said channels.

12. Apparatus according to claim 10 or 11, wherein the or each said longitudinal projection is straight and parallel to the axis of rotation.

13. Apparatus according to any one of claims 5 to 12, wherein said inner surface of said tube is of circular cross-section.

14. Apparatus according to any one of claims 5 to 13, wherein said outer surface of said member has multiple faces thereto.

15. Apparatus according to claim 14, wherein said outer surface is of regular polygonal cross-section.

16. Apparatus according to any one of claims 5 to 15, wherein said surfaces of said tube and member are respectively of uniform size and shape along the length thereof.

17. Apparatus according to any one of claims 5 to 15, wherein said surfaces of said tube and member have cross-sections which change along the length thereof.

18. Apparatus according to claim 17, wherein said tube and member are conically tapered.

19. Apparatus according to any one of claims 5 to 18, including means for heating or cooling the tubes.

20. Apparatus according to any one of claims 5 to 19, including a mixing chamber connected to said inlet for pre-mixing the reactants.

21. Apparatus according to claim 5, substantially as hereinbefore described with reference to the accompanying drawings.

22. A method according to claim 1, substantially as hereinbefore described with reference to the accompanying drawings.

23. A method according to any one of claims 1 to 4, when performed using the apparatus of any one of claims 5 to 21.

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